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# TECHNOLOGY DEVELOPMENT FOR TRANSITION METAL-RARE EARTH HIGH-PERFORMANCE MAGNETIC MATERIALS

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**TECHNOLOGY DEVELOPMENT FOR TRANSITION METAL-RARE  
EARTH HIGH-PERFORMANCE MAGNETIC MATERIALS**

**J. J. Becker**

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## ABSTRACT

The angular dependence of the behavior of magnetization reversal discontinuities in single-particle samples was studied in an effort to obtain more information about the nature of the imperfections responsible for the discontinuities. In some cases a  $1/\cos\theta$  dependence is observed, as would be expected for a pinned domain wall fragment. In other cases the angular dependence is flatter and sometimes unsymmetrical. Preliminary experiments indicate that study of angular and temperature variation of nucleation fields together should be very fruitful. It has been found possible to raise the coercive force of  $\text{Co}_{17}\text{Sm}_2$ , potentially a very high-performance permanent magnet material, by chemical treatment. Directionally solidified ingots of  $\text{Co}_5\text{Sm}$  have been prepared for microscopic studies. While preferred growth transverse to the c-axis seems to predominate, one large grain near the beginning of the ingot showed a classic high-anisotropy transverse domain pattern. Quantitative metallography has revealed no consistent variation of the proportion of samarium-rich phases with heat treatment. The minor impurities that might interfere with wet chemical analysis have been identified. It has been found that present processing techniques for sintered magnets do not substantially alter the composition of the starting material. Electron diffraction studies have identified the phases present in some sintered and heat-treated materials. Careful optimization of all processing procedures has resulted in a Co-Sm magnet with an energy product of 23.4 mGCe. Co-Pr-Sm magnets prepared from calcium-reduced oxide powders have shown an energy product of 25 mGOe. Measurements of  $B_r$  and of magnetization at various fields emphasize the importance of alignment in influencing magnetic properties.

## FOREWORD

This report describes work carried out in the Metallurgy and Ceramics Laboratory of the General Electric Research and Development Center, Schenectady, New York, under USAF Contract No. F33615-70-C-1626, entitled "Technology Development for Transition Metal-Rare Earth High-Performance Magnetic Materials." This work is administered by the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, J.C. Olson (AFML/LPE), Project Engineer.

This Second Semiannual Interim Technical Report covers work conducted during the period 1 January - 30 June 1971. The principal participants in the research are J.J. Becker, J.D. Livingston, J.G. Smeggil, D.L. Martin, and L. Valentine. The report was submitted by the author in July 1971.

The contractor's report number is S-71-1119.

This technical report has been reviewed and is approved.

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# TECHNOLOGY DEVELOPMENT FOR TRANSITION METAL-RARE EARTH HIGH-PERFORMANCE MAGNETIC MATERIALS

## I. INTRODUCTION

This is the second semiannual interim technical report for Contract No. F33615-70-C-162<sup>c</sup>, covering the period 1 January 1971 through 30 June 1971. The objective of this work, as set forth in Exhibit A of the contract, is to develop the technology of high-performance transition metal-rare earth magnets for critical applications. High-performance permanent magnets are defined in this context as those having remanences greater than ten thousand gauss and permeabilities of very nearly unity throughout the second and into the third quadrants of their hysteresis loop. Such technology is to be developed through 1) studies of the origin of the intrinsic coercive force in high-anisotropy materials, 2) development of information on phase equilibria in these systems, and 3) identification and investigation of new materials. The progress that has been made during the period covered by this report is described below under these three major headings.

## II. FUNDAMENTAL STUDIES OF THE ORIGIN OF THE COERCIVE FORCE IN HIGH-ANISOTROPY MATERIALS

### 1. Magnetization Studies in Single Particles (J. J. Becker)

The coercive force of cobalt-rare earth materials appears to be controlled by structural irregularities acting as nuclei of reverse magnetization. One way to learn more about the detailed nature of these irregularities is to analyze the magnetic hysteresis behavior of single particles, whose behavior is controlled by a relatively small number of defects. The First Semiannual Interim Technical Report (AFML TR-71-31) describes how hysteresis loops showing a number of characteristic magnetization jumps can be analyzed as the sum of loops of a simple type, indicating that different portions of a particle may act magnetically independently. Some experiments have since been done in which the angular dependence of the field for a given magnetization jump, after magnetizing in the easy direction, was measured. Many different types of nucleation or pinning sites responsible for magnetization "jumps," and therefore ultimately for the coercive force, can be envisaged. Different types of structural imperfections would show different dependence of jump field on various parameters. The dependence of jump field on angle might be expected to vary anywhere from a  $1/\cos\theta$  dependence (Kondorsky) to a sharp peak at  $\theta = 0^\circ$  (Stoner-Wohlfarth). If the reversal process is the unpinning of a stuck fragment of wall, a  $1/\cos\theta$  dependence would be expected--since only the resolved field in the easy direction is effective in producing a force on the  $180^\circ$  wall. If the wall is considered to be driven out entirely when the sample is saturated, it is considerably less clear what the details of the renucleation mechanism might be.

Reich, Shtrikman, and Treves<sup>(1)</sup> showed that the coercive force of some orthoferrite single crystals followed an exact  $1/\cos\theta$  dependence on angle. They point out that since  $H_C \ll K/M$ , large-scale rotation does not take place. "Thus," they say, "only the component of the external field parallel to the easy axis of magnetization affects the magnetization reversal process. The situation fits in with a picture in which the magnitude of the coercive force is governed by the nucleation of a  $180^\circ$  domain wall." The argument seems right for wall motion, but why should it also be true for nucleation? If a wall is created, local rotation must take place in some way, influenced by the local structure of the nucleating site. It seems very remarkable that they found a  $1/\cos\theta$  dependence for the nucleation process itself.

One might try to classify the various types of imperfections that might be operative in controlling coercive force, somewhat as follows:

(1) Purely geometrical. That is,  $M$  is uniform, but local fields are produced by the pole distributions on surface projections or indentations or internal voids or cracks. Nonmagnetic inclusions would be of this class, but there might be lattice strain associated with them as well.

(2) Lattice deformation. Elastic deformation, as around an inclusion, would add a local strain-magnetostriction anisotropy, whose magnitude would be unknown in these materials, in the absence of any information on their magnetostriction coefficients. Plastic deformation, as by mechanical processing, in which the lattice is disrupted, might well drastically affect the local anisotropy and perhaps also  $M$ .

(3) Composition variations. These can be inhomogeneities resulting from preparation, as for example from a peritectic reaction. They can result from local oxidation or volatilization of a component. They can also be associated with a discrete two-phase structure. In all cases the local  $M$  and  $K$  will gradually or suddenly change. The orientation of the local  $K$  axis relative to the rest of the material may also be important.

Any of these types of imperfections could conceivably act as either pinning centers or true nuclei. In the former case, a  $1/\cos\theta$  dependence would be expected; in the latter there would appear to be many possibilities. One experimental method that should be very useful in gaining more information about nuclei is the measurement of jumping fields or coercive force as a function of temperature. If the nucleus is a bit of second phase, with different  $M$ ,  $K$ , and Curie temperatures, the temperature dependence should in general be much the most pronounced. Preliminary experiments indicate that this is indeed the case. For example, a single-particle sample of  $\text{Co}_5\text{Sm}$ , after magnetization in 21 kOe, reversed its magnetization in two successive jumps of about the same size at 1200 Oe and 1600 Oe on each side of the hysteresis loop. After magnetization in 21 kOe at room temperature, it was then cooled to liquid nitrogen temperature and the demagnetization curve measured. In this case the result

was a rectangular hysteresis loop in which reversal took place in a single jump at 3800 Oe on each side. This line of investigation will be continued and should provide a powerful analytical tool.

The results of some room-temperature measurements of the angular dependence of jump field are shown in Fig. 1. The sample was a single particle of Co<sub>5</sub>Sm about 50 $\mu$  in average diameter, mounted in paraffin and aligned in a field. After saturation in 21 kOe, it displayed several reproducible jumps. The behavior of two of these is shown in the figure. For each measurement, the sample was magnetized in 21 kOe at 0°, i.e., in the easy direction, then rotated in one sense or the other and the reverse field increased until the jump occurred. The remarkable thing about these results is that the angular behavior is not symmetrical. The  $1/\cos\theta$  dependence is followed for one sense of rotation but not for the other. Possible explanations for this behavior are still in the realm of speculation.

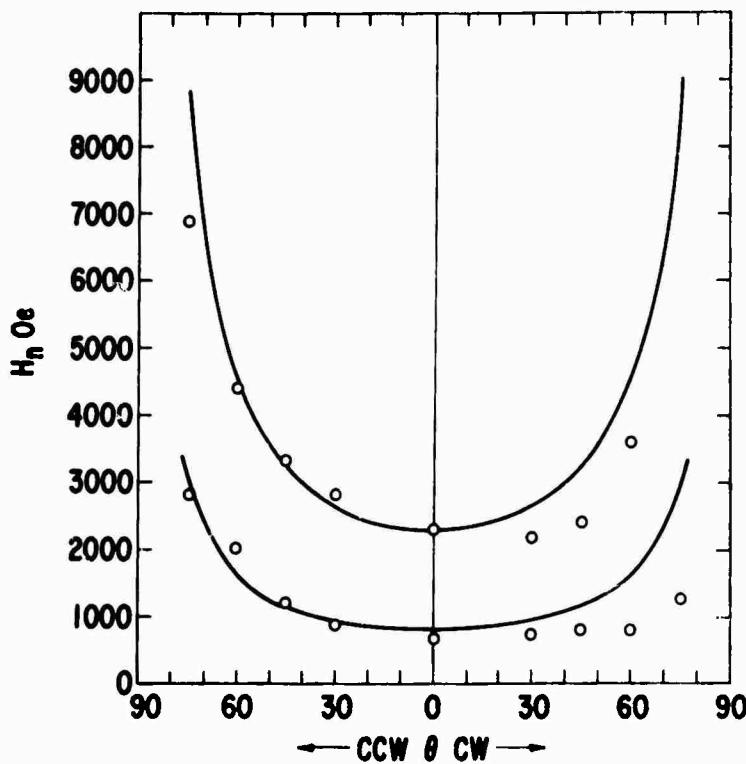


Fig. 1 Angular dependence of the applied fields  $H_n$  at which two magnetization jumps occur in a Co<sub>5</sub>Sm particle. The particle was first magnetized in 21 kOe in the  $\theta = 0$  direction, then rotated clockwise (CW) or counterclockwise (CCW), and the reverse fields at which the jumps occurred were measured. The solid lines are plots of  $H_n = H_n(0)/\cos\theta$ .

It might seem that the total field, rather than just the applied field, should be considered. However, whatever the local internal fields are, they remain constant with  $\theta$ . If, for example, the additional field needed to get a stuck wall fragment loose is  $H_a$  at  $0^\circ$ , the local fields will not vary with angle and the additional field at the angle  $\theta$  will still be  $H_a/\cos\theta$ .

A sample of high-anisotropy material in which easy wall motion is dominant should show Kondorsky ( $1/\cos\theta$ ) behavior. Low-coercive-force Co<sub>5</sub>Y shows a good  $1/\cos\theta$  behavior for  $H_c$  if the particles are not larger than about  $50\mu$ . Larger particles often consist of more than one grain. Since these are not perfectly aligned in the original casting, the effect, as can easily be seen, is to flatten out the angular dependence.

## 2. Effect of Chemical Treatment on Co<sub>17</sub>Sm<sub>2</sub> (J. J. Becker)

The compound Co<sub>17</sub>Sm<sub>2</sub> is a potentially high-performance material as defined in the work statement. Its saturation magnetization  $4\pi M_s$  is 12030 gauss,<sup>(2)</sup> so the energy product could be greater than 36 mGOe. It has a high easy-axis anisotropy<sup>(3)</sup> but has shown only low coercive forces when ground into particles in the usual way. It has previously been shown<sup>(4)</sup> that chemical smoothing of the particle surface, or alternatively attack by a dilute acid, could greatly raise the coercive force of Co<sub>5</sub>Y and Co<sub>5</sub>Sm. The coercive force of Co<sub>17</sub>Sm<sub>2</sub> can also be increased by treatment in either a chemical polishing solution or a dilute acid solution. This has been demonstrated in two samples. While both of them are nominally Co<sub>17</sub>Sm<sub>2</sub>, chemical analysis showed that one, sample MH577, contained 75.7 wt % Co, and the other, MH506, 77.0 wt % Co. Both showed the effect, and since they straddle the stoichiometric composition of Co<sub>17</sub>Sm<sub>2</sub>, which is 76.9 wt % Co, it appears that the effect is truly a property of that phase.

The results are summarized below; the numbers are intrinsic coercive forces:

	MH577	MH506
As ground to -325 mesh	240	310
5 sec in chemical polishing solution*	620	540
10 sec in chemical polishing solution*	870	580
20 sec in chemical polishing solution*	800	495
10 sec in 2% HNO <sub>3</sub>	320	325
30 sec in 2% HNO <sub>3</sub>	550	550
60 sec in 2% HNO <sub>3</sub>	605	390

\*3 parts HNO<sub>3</sub>, 1 part H<sub>2</sub>SO<sub>4</sub>, 1 part H<sub>3</sub>PO<sub>4</sub>, and 5 parts CH<sub>3</sub>COOH by volume.

These values of coercive force are not yet as high as they need to be, but the fact that  $H_c$  can be increased by a factor of about 5 in this potentially high-performance material appears to be significant.

### **3. Microscopy and Domain Structure of Alloys and Finished Magnets (J. D. Livingston)**

---

Directional-solidification experiments have been initiated for the purpose of producing large-grain samples for domain studies. Several crucibles were prepared of "Yttralox," a special high-density yttria developed by General Electric. These crucibles were 3.5" long, 0.275" I.D., and 0.462" O.D. Portions of a cast ingot of cobalt--34 wt % samarium--were melted in an Yttralox crucible under an argon atmosphere, and solidified at a rate of 2 inches per hour. Although reaction between sample and crucible was slight, adherence of the sample to the crucible walls required destruction of the crucible to remove the sample.

Transverse and longitudinal sections of the sample were prepared and studied in polarized light. Near the beginning of the ingot, one large grain in transverse section showed the domain structure typical of a surface normal to the c-axis, indicating that in this grain the c-axis was the growth direction. This structure is shown in Fig. 2. However, near the end of the ingot the domain structure appeared to be predominantly transverse to the growth direction, suggesting that the preferred growth direction is transverse to the c-axis. Some dendritic structure was also observed. Study of the phase diagram suggests that the dendrites may be of the  $Co_{17}Sm_2$  phase, but positive identification must await planned x-ray and microprobe studies.

Quantitative metallography by point-count techniques has been applied to sintered Co-Sm magnets after heat treatment at various temperatures. The goal was to determine whether any change in the proportion and distribution of the phases present could be observed and correlated with domain structures and magnetic properties. No consistent variation of the proportion of samarium-rich phases has been observed. More study is required to determine if a variation in the proportion of oxide phase exists and is significant.

An investigation by transmission electron microscopy of the internal microstructure of Co-Sm after various heat treatments is under way.

## **III. CHARACTERIZATION OF COBALT-RARE EARTH MATERIALS**

### **1. Chemical Analysis (J. G. Smeggil)**

The problems associated with the use of wet chemical techniques in the analysis of Co-Sm materials have been eliminated by taking into account both the presence and the possible interfering effect of minor impurities. It has been found, for example, that the concentration of Al, although at a very low

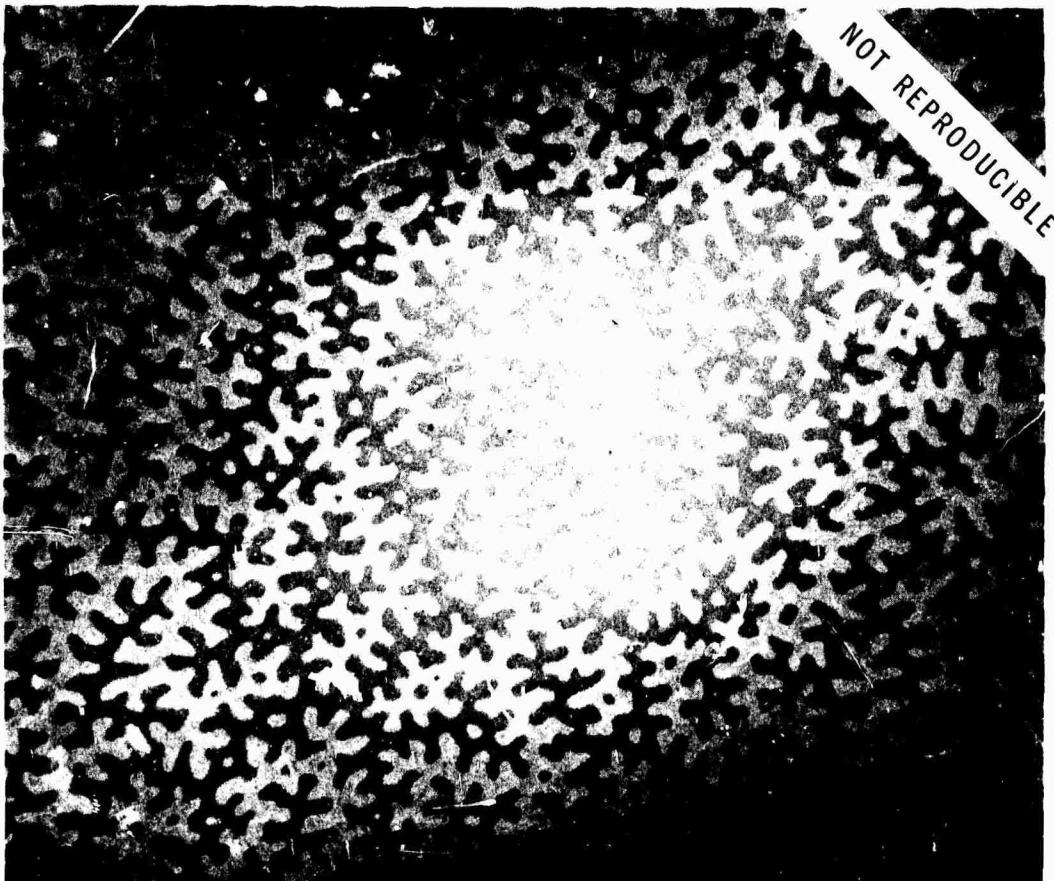


Fig. 2 Magnetic domain structure of transverse section of directionally solidified Co-Sm ingot. Polarized light. 500X

value of about 0.065 wt % potentially can exhibit a significant effect on the rare earth analysis. If the Al is analyzed as moles of Sm, then Al at a concentration of 0.065 wt % will be reported as  $(150/27 \times 0.065)$  0.35 wt % Sm. The source of the contaminating Al has been identified as the  $\text{Al}_2\text{O}_3$  crucible in which the Co-Sm alloys are initially made.

The presence of Ni has been determined in the starting Co material, in amounts varying from 0.054 to 0.45 wt %. The variation in the amount of Ni is associated with different sources of the Co material.

Oxygen has been found to occur in sintered Co-Sm materials at concentrations of about 0.30 wt %.

Whether analyses for Al, Ni, and O are required each time the relative amounts of Co and Sm are desired is not known and is under investigation at the present time.

One specific purpose of acquiring accurate chemical analyses of the cobalt-rare-earth compounds under investigation in this program is to provide suitable standards for a rapid and reliable instrumental analytical technique. A standardized instrumental technique, such as x-ray fluorescence, will allow the correlation of composition and properties in a more accurate and routine fashion than is presently possible.

To determine if present processing techniques for liquid-phase sintered materials are such that the material is depleted by preferential Sm vaporization or is degraded by substantial oxidation, a chemical analysis was undertaken of a bar of Co-Sm material pressed from the powder. The bar was cut into two pieces and then one of the pieces was sintered at about 1100°C for one hour. The results of this analysis are listed in Table I.

TABLE I

	<u>Pressed Powder</u>	<u>Sintered Rod</u>
Wt % Co	63.54 ± 0.03	63.54 ± 0.03
Wt % Sm	35.55 ± 0.03	35.61 ± 0.03
Wt % O	0.29 ± 0.03	0.34 ± 0.03

These results indicate that the present processing techniques do not substantially alter the composition of the starting material. The initial Co contained 0.45 wt % Ni, and the sintered material probably contained about 0.26 wt % Al.

## 2. Structure Analysis (J.G. Smeggil)

Electron transmission studies of Co-Sm sintered materials have resulted in identifying  $\text{Co}_5\text{Sm}$ ,  $\text{Co}_7\text{Sm}_2$ ,  $\text{B-Sm}_2\text{O}_3$ , and  $\text{C-Sm}_2\text{O}_3$ . Only the cubic  $\text{C-Sm}_2\text{O}_3$  was observed in material heat treated at about 1100°C for 1/2 hour, while only the  $\text{B-Sm}_2\text{O}_3$  was observed in a sample heated to 1200°C for 7 1/2 hours. The identification of the various phases was made on the basis of both the lattice parameters and the diffraction symmetry of the various phases with particular emphasis placed on the low angle reflections.

## IV. ALLOY DEVELOPMENT

### 1. Co-Sm Magnet Optimization (D.L. Martin)

It has been demonstrated that by careful optimization of all processing variables, including alignment, sintering, and heat treatment, a Co-Sm magnet with an energy product of 23.4 mGOe can be made.<sup>(5)</sup> This is 84% of the theoretical limit of  $(4\pi J_s)^2/4$ , and indicates how close to the ultimate energy product it is possible to achieve.

The sample was made by the liquid-phase sintering process in which a Co<sub>5</sub>Sm base metal alloy powder was blended with a 60 w/o Sm-Co alloy powder prior to alignment and sintering.<sup>(5)</sup> The powder was aligned in a 60 kOe field, followed by hydropressing at 200,000 psi prior to sintering in a purified argon atmosphere at 1135°C. A treatment at 850°C was given after sintering. The post-sintering treatment has been found to be an effective method of improving the coercivity.<sup>(6)</sup> The B:H curves in Fig. 3, measured after magnetizing at 60 kOe, show the improvement resulting from the post-sintering treatment, and also from sintering at a higher temperature.

The data for the Co-Sm magnet are listed in Table II. The saturation value of 10.5 kG is somewhat higher than the value of 9.65 kG which is usually quoted.<sup>(7)</sup> As a result of its higher saturation, this sample has a theoretical limit of 27.7 mGOe. As noted previously the (BH)<sub>max</sub> value of 23.4 mGOe represents an attainment of 84% of the theoretical value.

The higher value of  $4\pi J_S$  obtained for this sample is believed to be related to a high degree of alignment together with powder consisting essentially of single-crystal grains. Multicrystalline grains are harmful because they cannot be aligned to as high a degree as single-crystal grains. A detailed discussion of alignment is given in Section 3.

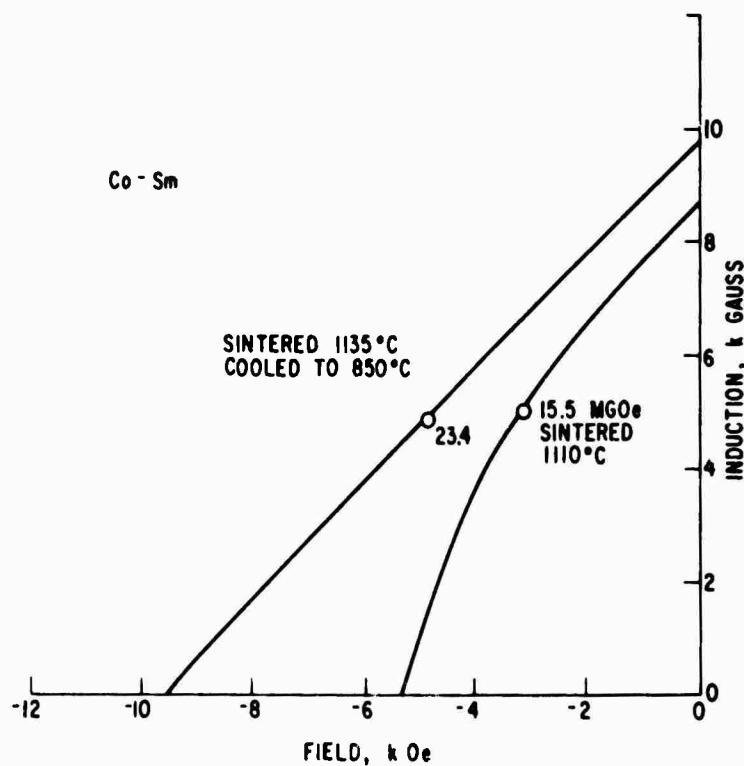


Fig. 3 Variation of B vs H curve characteristic of a nominal 63 wt %Co-Sm alloy with sintering temperature and post-sintering heat treatment.

noteworthy, the  $B_{H_C}$  value of 10,000 Oe is particularly significant because of the importance of high coercivity for many applications. A summary of data for the alloys studies is given in Table III.

### 3. The Alignment Factor (D. L. Martin)

The role of alignment factor and its effect on magnetic properties has been studied in detail. A series of Co-Sm specimens with different degrees of alignment were prepared by varying the prealignment packing of the powder and the magnitude of the alignment field. The first quadrant magnetization curves and the second quadrant demagnetization curves were measured.

Each sample was exposed to a series of increasing magnetizing fields and the magnetization measured by a method described previously.<sup>(11)</sup> After each measurement, the sample was taken out of the field and the open circuit magnetization was measured. For a long bar, the  $4\pi J$  value so measured is a close approximation of the  $B_r$  value. Thus, data was obtained on

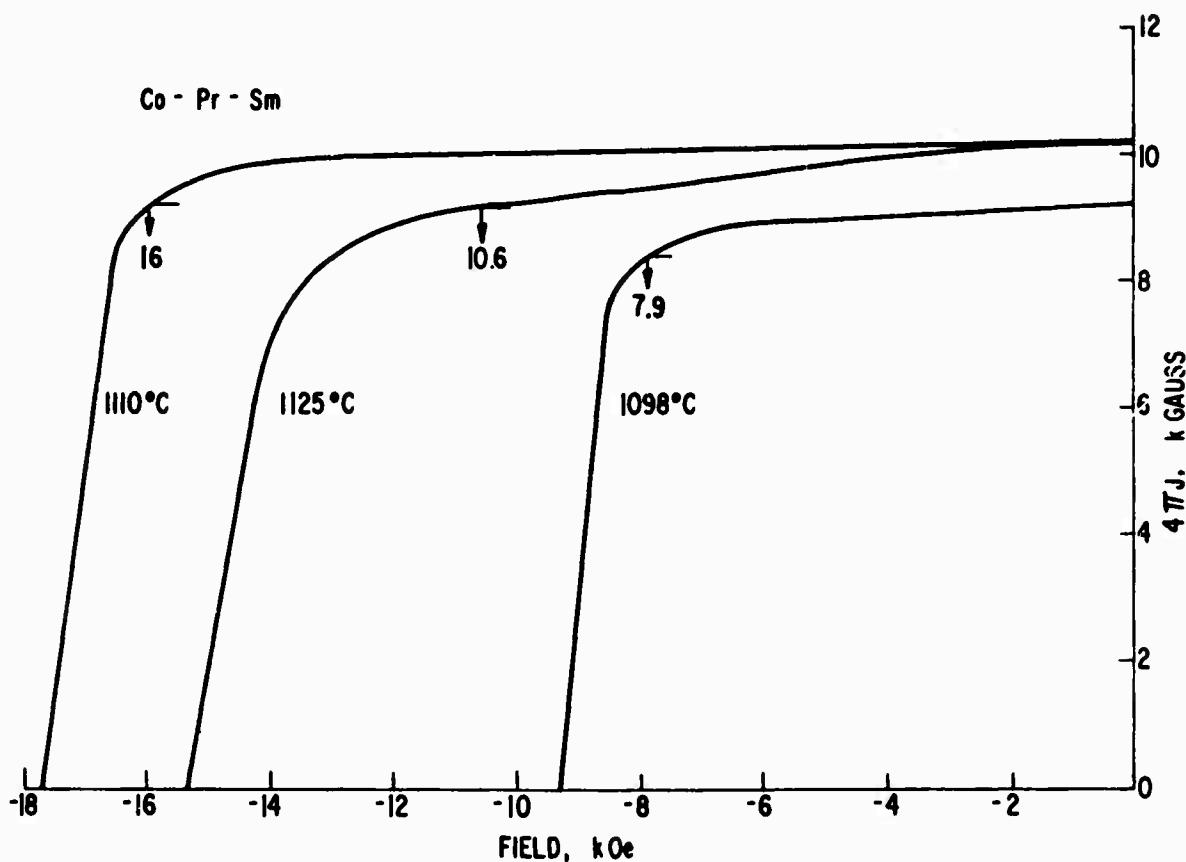


Fig. 5  $4\pi J$  vs  $H$  curves for Co-Pr-Sm magnet samples sintered at different temperatures.

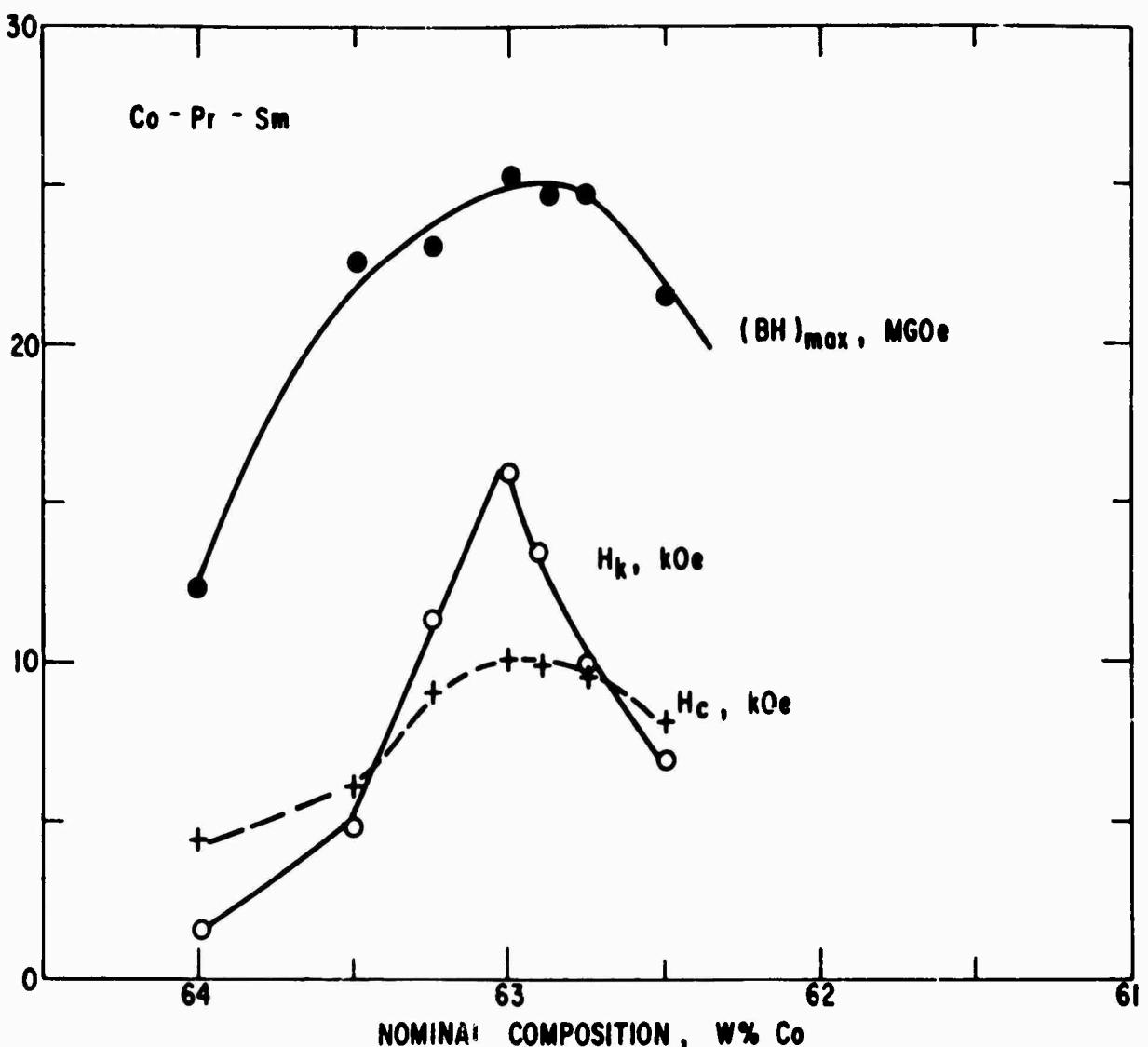


Fig. 6 Variation of magnetic properties with blend composition. The alloys were made by blending a nominal 67.3% Co, 24.6% Pr, 8.1% Sm base-metal powder with a 60% Sm, 40% Co additive powder.

the variation of  $4\pi J_s$  and  $B_r$  with peak magnetizing field. The results are plotted in Figs. 8 and 9.

The alignment factors listed were obtained by the following relation:

$$A' = \frac{B_r(100)}{pB_s}$$

where  $B_r(100)$  is the residual induction value obtained after magnetizing at

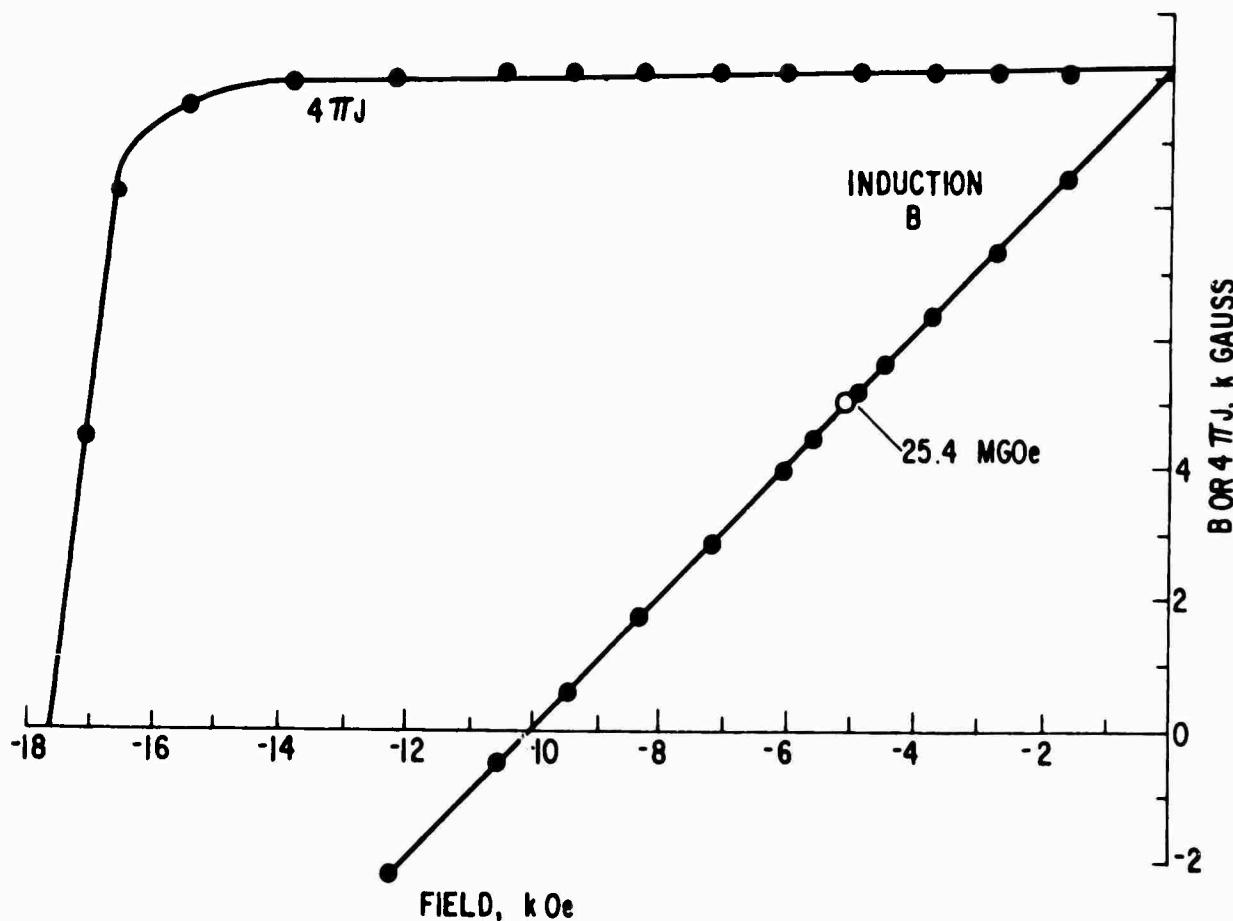


Fig. 7 Demagnetization curves for a high-performance Co-Pr-Sm magnet.

100 kOe,  $p$  is the packing fraction, and  $B_s$  is the saturation value of the alloy used in the study. The saturation was calculated from the  $B_r$  measured for a random unaligned sample with a known packing fraction as follows:

$$B_s = \frac{2 \times B_r}{P} \text{ (random)} \quad \text{or 10,000 gauss}$$

Note that  $A'$  differs from the alignment factor  $A_{60}$  given in Table II. The results in Fig. 8 show the change in  $B_r$  with alignment factor and magnetizing field. Above a field of 60 kOe, the  $B_r$  value for each sample does not change noticeably. In contrast, the magnetization,  $4\pi J$ , for the samples with a poor degree of alignment is still increasing at 100 kOe (Fig. 9). It is quite clear from these two figures that alignment is an important factor to control if optimum properties are to be realized. The demagnetization results listed in Table IV give additional emphasis to the importance of alignment in alloy

TABLE III  
Summary of Magnetic Data for Co-Pr-Sm Alloys

Nominal w/o Cobalt	Sintering Treatment °C	$4\pi J_s$ kGauss	$B_r$ kGauss	$H_c$ kOe	$H_k$ kOe	$j^H_c$ kOe	$(BH)_c$ mOe	$(BH)_{max}$ mOe	Density g/cc	P	$A_{40}$
62.5	1 hr. 1118°	10.6	9.7	6.8	5.4	10.9	21.6	8.05	95	95	97
62.75	1 hr. 1110°	11.0	10.1	9.5	9.8	12.4	24.7	8.00	94	94	98
62.9	1 hr. 1115°	10.7	10.1	9.9	13.6	16.8	24.8	8.10	95	95	98
63.0	1 hr. 1098°	10.3	9.2	8.2	7.9	9.3	20.3	7.92	93	93	96
	1 hr. 1105°	10.9	10.0	9.9	14.7	16.8	24.8	8.02	94	94	98
	1 hr. 1110°	11.1	10.2	10.0	16.0	17.6	25.4	8.07	95	95	98
	1 hr. 1115°	11.0	10.2	9.7	14.2	18.9	25.1	8.07	95	95	98
	1 hr. 1120°	11.0	10.2	9.9	14.0	16.3	25.2	8.07	95	95	98
	1 hr. 1125°	10.9	10.2	9.3	10.6	15.3	24.0	8.07	95	95	98
63.0	1 hr. 1110°	10.8	10.1	9.5	10.0	13.2	24.8	8.12	96	96	98
63.0	1 hr. 1119°	10.8	10.0	9.6	11.8	14.7	24.6	8.10	95	95	98
63.25	1 hr. 1120°	10.7	9.9	9.0	9.6	13.2	23.1	8.07	95	95	98
63.5	1 hr. 1120°	11.0	10.1	6.0	4.7	7.1	22.5	8.02	94	94	97
64.0	1 hr. 1125°	11.3	10.0	1.7	1.4	1.8	12.3	8.02	94	94	93

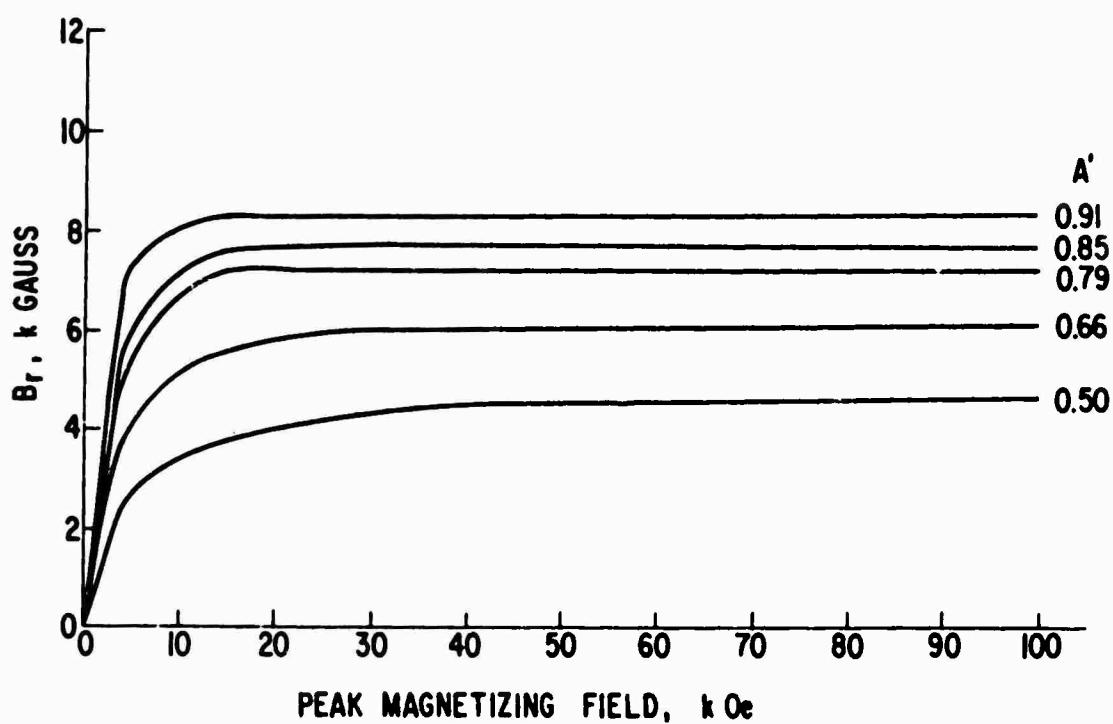


Fig. 8 Variation of  $B_r$  with magnetizing field for a series of Co-Sm samples with different degrees of alignment.

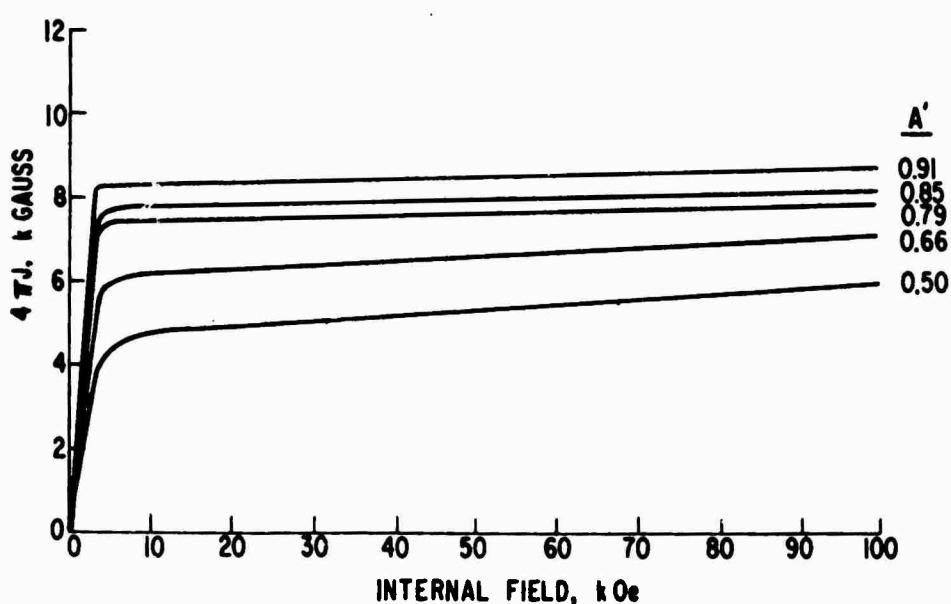


Fig. 9 Variation of  $4\pi J$  with magnetizing field for a series of samples with different degrees of alignment.

TABLE IV

Demagnetization Results on a Series of Cobalt-Samarium Magnets  
with Varying Alignment Factors. The Samples were Magnetized  
at 100 kOe Prior to Testing

No.	Nominal w/o Cobalt	Treatment (°C)	$4\pi J_S$ (kG)	$B_r$ (kG)	$H_c$ (kOe)	$H_k$ (kOe)	$jH_c$ (kOe)	$(BH)_{max}$ (mGOe)	Density (g/cc)	P	A'
A	37	1 hr 1120	6.07	4.56	-4.2	-4.8	-25.1	5.1	7.82	0.909	0.50
B	37	1 hr 1120	7.42	6.08	-5.2	-4.6	-22.2	8.7	7.83	0.911	0.67
C	37	1 hr 1125	8.61	7.31	-5.4	-3.4	-15.0	11.3	7.74	0.90	0.81
D	37	1 hr 1125	8.93	7.76	-5.7	-4.2	-16.9	13.3	7.79	0.906	0.86
E	37	1 hr 1125	9.42	8.28	-5.9	-4.7	-13.4	15.3	7.80	0.907	0.91

development studies. Thus, the energy product varied from 5 to 15 mGOe as the alignment increased from a random condition to over 0.9.

A surprising result of this study is the strong dependence of the magnetization measured at 100 kOe on the alignment. This is shown in Fig. 10 as well as in Fig. 9.

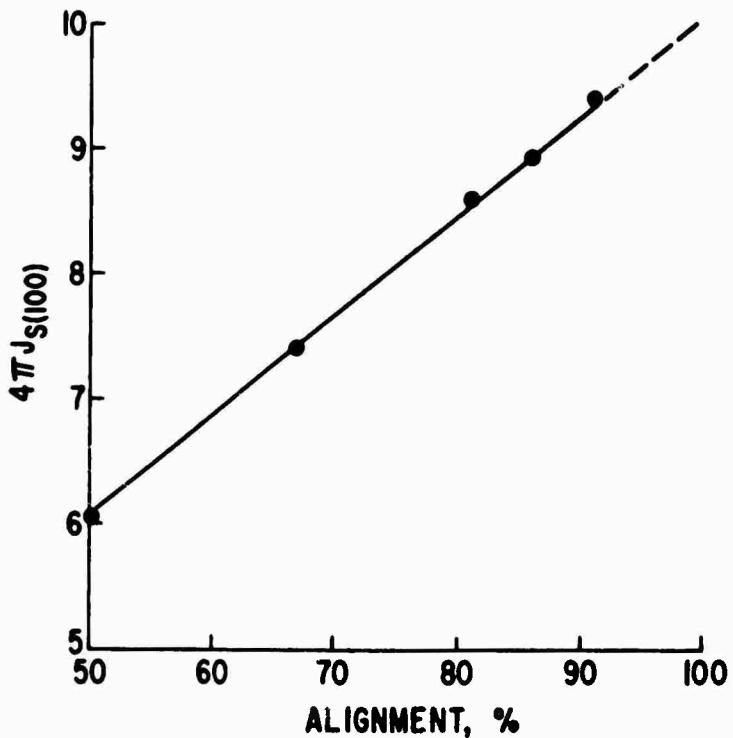


Fig. 10 Variation of magnetization,  $4\pi J_{100}$ , with degree of alignment, A'.

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